LONG TERM DURABILITY TESTING OF POLYMER COMPOSITE MATERIALS

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SUMMARY: This paper discusses the results of the long term polymer composite durability testing program conducted by the Aerospace Engineering department at California State University, Long Beach under sub-contract to The Boeing Company. This testing program was part of the NASA sponsored High Speed Research (HSR) composite durability program. The objective of the program was to develop a composite material system that could withstand the operational requirements of a 300 seat commercially viable aircraft known as the High Speed Civil Transport (HSCT) capable of cruising at speeds of Mach 2.4. In order to meet the durability requirements, a representative structural thermo-mechanical loading profile was taken from trans-pacific flight of 4.25 hours. The entire test program was designed to run for one aircraft lifetime or 60,000 hours of real time fatigue testing. The results presented in this report are for the initial check-out run of 3,300 hours.

KEYWORDS: aircraft, durability, high temperature, polymer

ABSTRACT

This paper details the effort involved in evaluating two polymer composite material systems (one cyanate ester 954-2A and one polyimide K3B) undergoing long term thermo-mechanical fatigue testing. The loading profile simulates a trans-pacific HSCT flight in 4.25 hours which takes into consideration subsonic as well as supersonic flight conditions and typical maneuver loads. Initial test lab check out results (3,300 hours) for the two material systems indicated that the polyimide resin system has the potential for surviving the 60,000 hour testing requirement, while the cyanate ester exhibited major resin degradation and was withdrawn from the test program. The in-plane mechanical properties for the polyimide resin indicated no loss in strength however out-of-plane properties did exhibit some degradation which may be due to microcracking. Since the check out test program, the lab continues to run seven test frames accumulating over 7,000 hours per year per frame at 95% efficiency.

HSCT LONG-TERM DURABILITY REQUIREMENTS

The long-term integrity of high-temperature structure is ensured through the proper selection of materials that meet durability and damage tolerance design requirements. Accounting for material damage resistance and damage tolerance is a critical design requirement. FAR 25.571 states: no catastrophic structural failure shall occur throughout the operational life of the aircraft. Advisory Circulars AC 25.571-1 and AC 20-107A provide further guidance. The actual means for showing compliance must still be formulated by the aircraft manufacturer and submitted for approval by the FAA. The HSR program has designed a program to meet these requirements by exposing the materials to one aircraft's lifetime worth of testing while simultaneously developing methods to accelerate these tests.

The design life of the HSCT is 60,000 flight hours or 20,000 ground-air-ground flight cycles. Since there are only 8,760 hours per year, it would take over 7 years at 100% utilization to accumulate one-lifetime worth of aging. With stoppages for maintenance and repair this will extend the testing period to approximately eight years. One misconception however, is that the specimen must be undergoing thermal mechanical; fatigue without stoppage to provide a valid technical result. This is not the case, and the laboratory will be shut down for annual maintenance for items including oil and filter replacement.

REVIEW OF PREVIOUS WORK

A study of the long term behavior of polymer composites was conducted by Kerr and Haskins at GD/Convair under NASA Langley funding (1973-1987) (Reference 1). Their work included the long term evaluation of a polyimide resin system, HT-S/710. The HT-S/710 resin was thermally aged out to 50,000 at 450^{o}F (232^{o}C) where some decrease in tensile strength was found. Results at 25,000 did not exhibit any form of degradation thus the concluded that the operational use temperature of the HT-S/710 should not safely exceed 25,000 hours at 450^{o}F (232^{o}C). At the time, the authors concluded that current state-of-the-art (1970-1980) carbon/polyimides such as HT-S/710 should be limited to 450^{o}F (232^{o}C) when exposures are greater than 10,000 flight hours. Other resin systems were evaluated as part of the program but none were seen capable of temperatures in excess of 350^{o}F (177^{o}C).

HSCT FLIGHT PROFILE DEVELOPMENT

A thermal mechanical fatigue profile is required in order to accurately evaluate the long term durability of candidate materials for the HSCT. The mission profile used for aircraft sizing studies is the basis for the test profile. It includes a subsonic and a supersonic flight segment. The first 25 percent of the range is flown at Mach 0.95 to eliminate the sonic boom overland, while the remainder of the cruise segment is flown at Mach 2.4. This entire flight profile covers the maximum 5,500-n-mi (10,100 km) range of the aircraft. During flight, the HSCT structure will be exposed to two separate types of loading, the mechanical loads induced by flight and the thermal loads caused by aerodynamic heating.

The flight spectrum chosen for this program is based upon the temperature and loading profile experienced by the wing, where the temperatures are slightly higher than those seen by the fuselage (Figure 1).

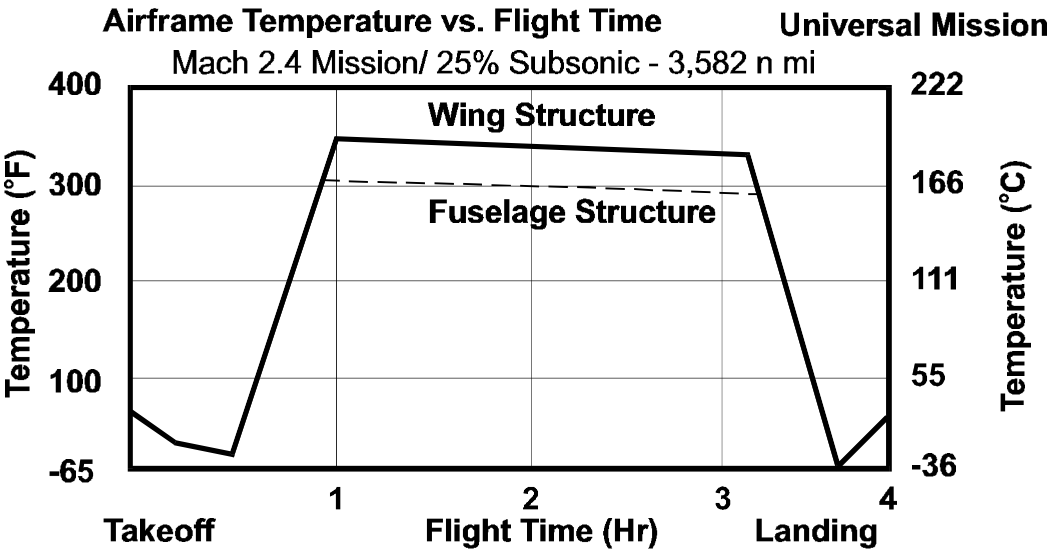

Fig. 1: HSCT temperature flight profile for long-term durability test

Once the baseline flight profile was developed, g-force calculations were taken into consideration. These data were generated using Boeing computer codes, which are based on atmospheric models developed by NASA, and Boeing commercial service experience on previous transport aircraft programs which include the flight maneuver and gust loads. The g-forces within $\pm$0.25 were truncated to reduce the number of low-level load inputs. Once these loads were generated, they were reduced to 40 independent loading points (the amount of RAM associated with each loading frame is only 5 kilobytes without the use of an independent computer). The final loading profile is shown in Figure 2.

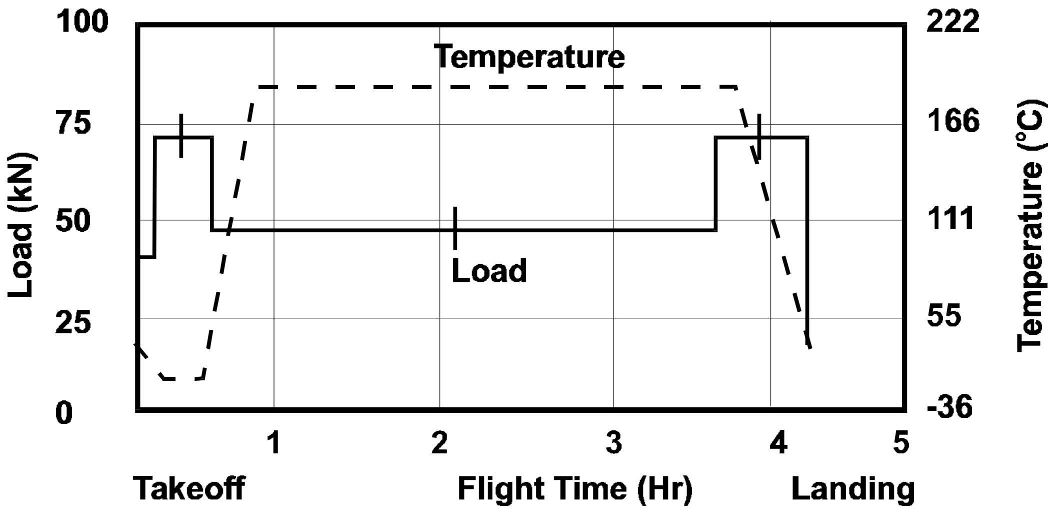

Fig. 3: Representative HSCT Thermo-mechanical loading profile

DETAILS OF CSULB LABORATORY SET-UP

In 1992, CSULB Long Beach Department of Aerospace Engineering initiated discussions with Instron Corporation to purchase ten load frames to the same specifications as those developed by NASA Langley Mechanics of Materials Branch. The CSULB laboratory consists of 10

Instron load frames. These include four - 22 kip (100 kN) load frames with thermal chambers, four - 55 kip (250 kN) load frames with thermal chambers, one 22 kip (100 kN) research station, and one 55 kip (250 kN) research station.

Eight of the load frames are supplemented with computer controlled Instron temperature chambers with temperature capability to 600°F (315°C). Convection heating is provided by electrical heating coils mounted on the back of the chamber. The chambers are operated at below ambient temperatures by use of a cryogenic liquid nitrogen which is supplied to the frames by Dewers of liquid nitrogen. The cryogen is sprayed into the chamber near the fan to ensure even distribution. When the desired temperature has been reached, the temperature controller regulates the liquid flow with a solenoid valve. The chambers also incorporate an exhaust port to allow waste gases to be safely ducted to a well-ventilated area for dispersal.

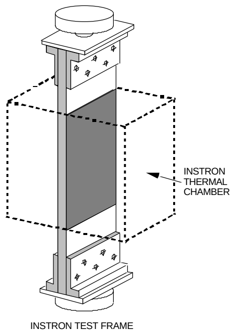

Fig. 3: Long-term durability test panel and thermal chamber

DETAILS OF PANEL GEOMETRY/TESTING

The panels used in the program are 51-inches (1.3 m.) long and 12 -inches (0.30 m.) wide (Figure 4). Each of the tension panels is 16 plies thick with a quasi-isotropic lay-up $[+45/-45/0/90]_{2S}$. The panel contains five staggered one inch (2.5 cm.) bolt holes at each end for loading. The panels are attached to L-grips and platens designed to hold the composite panels for long-term testing. The useful area that will be exposed to the thermal mechanical fatigue is 12-inch (0.30 m.) by 24-inch (0.60 m.). Test coupons will be excised from this section.

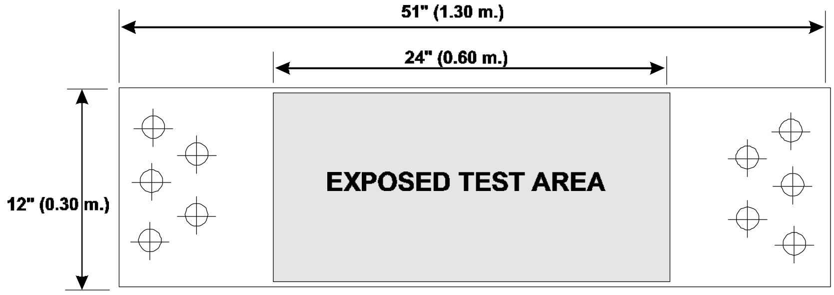

Fig. 4: Long-term durability test panel geometry

THERMO-MECHANICAL FATIGUE TESTING

The material systems chosen for long term durability evaluation included a thermoplastic polyimide K3B and a cyanate ester resin system 954-2A. The decision to select these materials was a result of a HSR round robbin test program which included over 15 resin systems. The thermoplastic polyimide material system K3B was chosen for its stability under long term isothermal aging, while the cyanate ester was chosen for its ability to degrade at an accelerated rate. It was hoped that the weaker cyanate ester material would degrade faster than the thermoplastic polyimide and offer researchers a glimpse at the future degradation of model materials to guide material development and correlate analyses. Both material systems were scheduled to undergo long term durability testing for the entire program's duration.

VARIATIONS IN LOADING PROFILE

Initially one thermoplastic polyimide (denoted in the test matrix as F-FOM1) panel was used to begin testing. The panel underwent 1,000 hours of thermo-mechanical testing and was subsequently removed for non-destructive evaluation. The panel was C-scanned and X-rayed. Both tests produced negative results.

Four additional thermo-mechanical fatigue tests were then initiated. Two thermoplastic polyimide panels were entered into the test program (F-FOM2 & F-TMF3). Two in-spec cyanate ester panels also entered the program at this stage (L-TMF2 & L-TMF3). The flight profile spectra for each panel varied slightly to examine the effect of temperature and peak loads on the degradation of the two material systems. All of these tests were limited to tension-tension fatigue. Two thermoplastic polyimide "rider" panels (F-TC1 & F-TC2) and one cyanate ester (L-TC1) were inserted into the thermal chambers and cycled between 0^{o}F (-17^{o}C) and 350^{o}F (177^{o}C) without mechanical loading. These three "rider" coupons (24 in. (0.60 m.) by 24 in. (0.60 m.)) were inserted into three separate thermal chambers by leaning them against the side of the oven's interior wall. The test matrix with the final amount of test hours accumulated on the initial test panels are shown in Table 1.

PANEL	MATERIAL	MAXIMUM LOADING KIPS (kN)	MAXIMUM STRAIN (MICRO IN/IN)	THERMAL CYCLE RANGE °F(^{o}C)	FINAL TEST HOURS
F-FOM1	POLYIMIDE K3B	27 (120)	3,000	0-350 (-17 - 177^{o}C)	3,370
F-FOM2	POLYIMIDE K3B	41 (182)	4,500	0-350 (-17 - 177^{o}C)	2,380
F-TMF3	POLYIMIDE K3B	22 (98)	2,450	0-440 (-17 - 226^{o}C)	2,346
L-TMF2	CYANATE ESTER 954-2A	22 (98)	2,450	0-350 (-17 - 177^{o}C)	2,367
L-TMF3	CYANATE ESTER 954-2A	22 (98)	2,450	350 (177^{o}C) (CONSTANT)	2,393
F-TC1	POLYIMIDE K3B	0	0	0-350 (-17 - 177^{o}C)	2,367
F-TC2	POLYIMIDE K3B	0	0	0-350 (-17 - 177^{o}C)	2,380
L-TC1	CYANATE ESTER 954-2A	0	0	0-350 (-17 - 177^{o}C)	2,367

Table 1: Initial Panel Test Log

RESULTS

Residual strength results shown in Figures 5 & 6 demonstrate the negligible loss in compressive and flexural strength for the thermoplastic polyimide material. The open hole compression strength results for thermoplastic polyimide are shown in Figure 5. While there appears to be a very slight decrease in average strength after aging, all of the mean values fall within one standard deviation of the unaged material. It should also be noted that the F-FOM1 and F-FOM2 panels were made from a different batch of material which had exceeded its shelf life by six months. Subsequent testing verified this factor to have little effect on the material properties.

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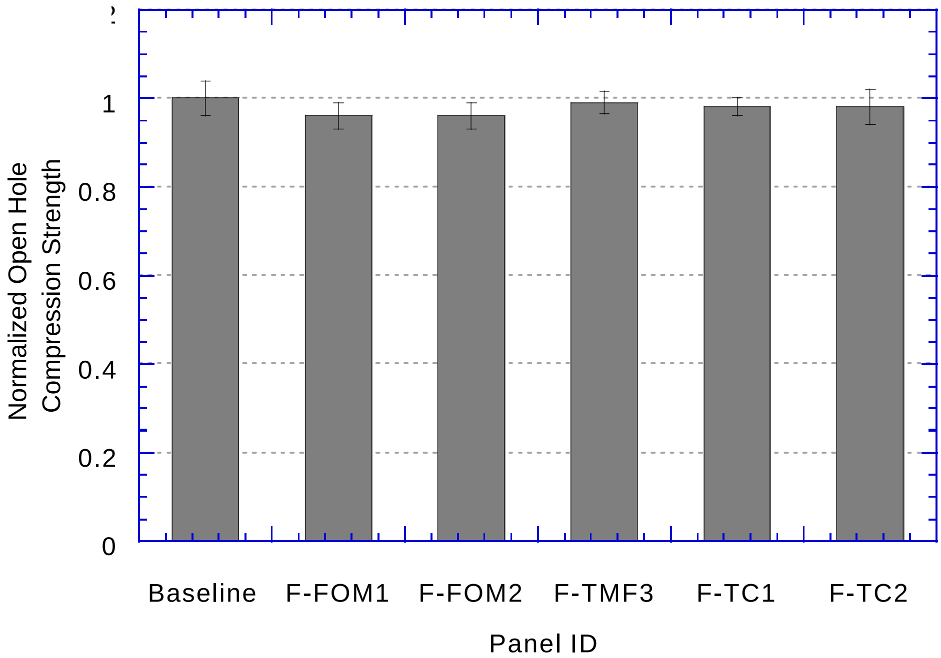

Fig. 5: Normalized Open Hole Compression Strength of Thermo-Mechanically Aged Polyimide K3B

The open hole tension strength results were similar to the compression results.. Again, very little change occurs after the material ages.

The results of the four point bend tests are shown in Figure 6. These results indicate a drop in flexural strength after aging for all of the conditions. The results from the F-FOM1 are significantly lower since the panel had an extra 1,000 hours of aging. F-FOM2 with 2,380 hours saw a degradation due to the higher load levels, twice as high as F-FOM-1, while F-TMF3 experienced the same amount of degradation as the purely thermally cycled panels F-TC1 & F-TC2 despite the loading and increased temperature range 0 to 440^{o}F (-17 to 226^{o}C) seen by F-TMF3. These initial results indicate that time is more of a degradation mechanism than increasing the temperature range. Further testing in the second phase of the program will confirm this effect. Micrograph inspection of the panels did however reveal some microcracks throughout the entire thickness of the laminate (Figure 7). Close monitoring of these cracks may indicate future degradation of the material system.

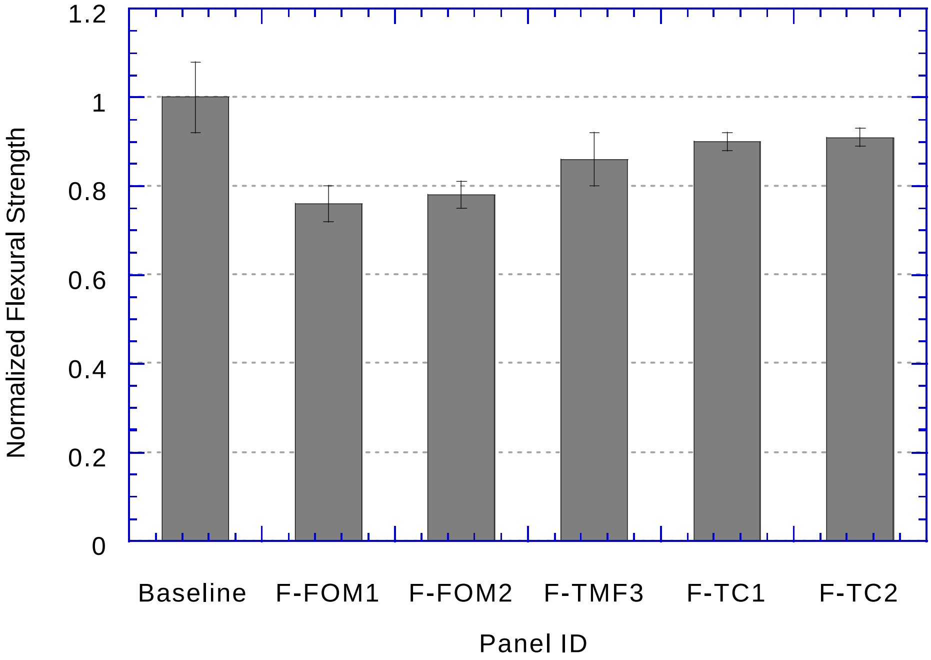

Fig. 6: Normalized 4 Point Bend (Outer Fiber) Strength of Thermo-Mechanically Aged Polyimide K3B

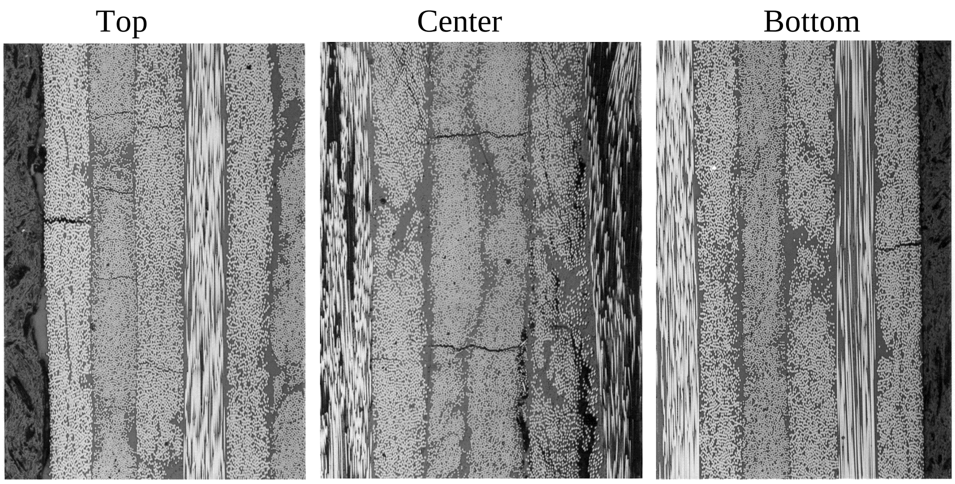

Fig. 7: Microcrack Detection in Aged Polyimide K3B after 3,300 hours (F-FOM1)

While the thermoplastic polyimide material did not demonstrate any large drop in residual strength in in-plane properties, the cyanate ester material did exhibit considerable degradation. At the conclusion of the final aging hours, the cyanate ester coupons had enough surface degradation from resin burn-off that bonding strain gages onto the specimens proved to be quite difficult. Micrograph inspection of the laminates revealed areas of resin matrix degradation (Figure 10). This degradation resulted in a much larger than expected scatter in the results. Figures 8 & 9 demonstrate the loss in compressive and flexural strength for this material system.

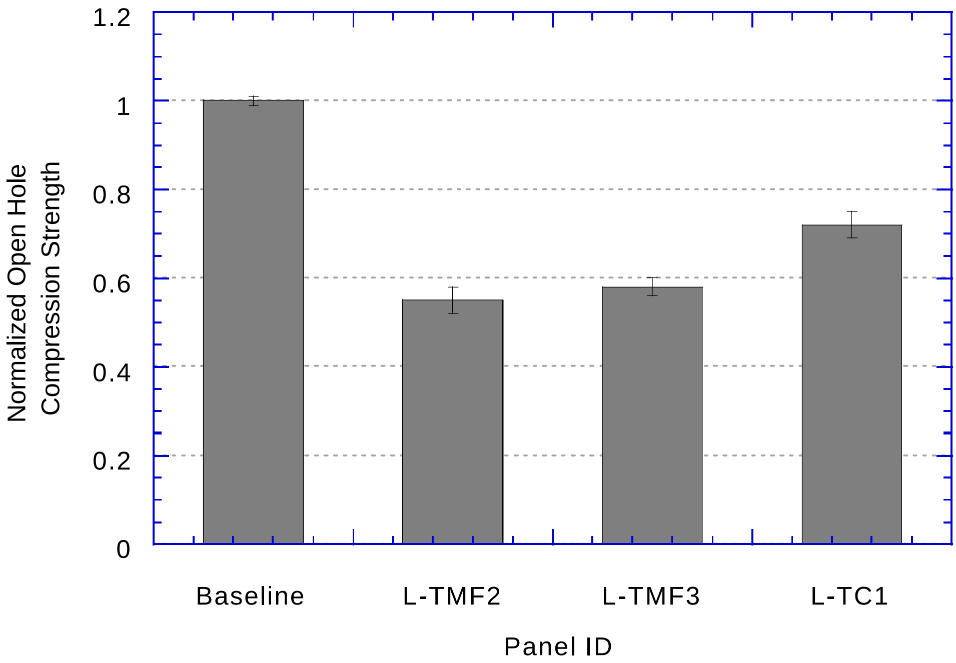

Fig. 8: Normalized Open Hole Compression Strength of Thermo-Mechanically Aged Cyanate Ester 954-2A

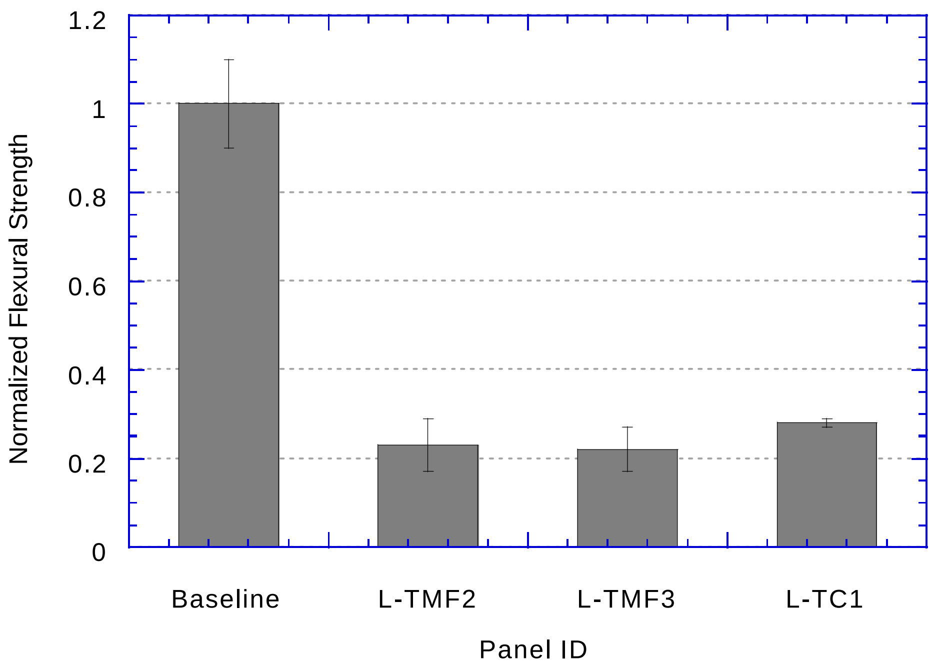

Fig. 9: Normalized 4-Point Bend (Outer Fiber) Strength of Thermo-Mechanically Aged Cyanate Ester 954-2A

Fig. 10: Resin Matrix Degradation of Cyanate Ester after 2,393 hours (L-TMF3)

Based upon mechanical property results, the TMF panels L-TMF2 and L-TMF3 exhibited a larger amount of degradation than the thermally cycled panels L-TC1. This considerable loss in material properties led to the removal of the cyanate ester system from the long term durability testing program. It was decided that the cyanate ester resin was degrading at a more rapid rate than expected and the degradation process was considerably different than the polyimide resin. Thus the second set of long term durability tests did not include the cyanate ester resin system.

FUTURE WORK

After completing the first set of tests, the CSULB lab underwent some modifications. Initially, each frame's cooling system was supplied via 50 gallon (0.19 m^3) Dewers. Each frame consumed approximately 40 gallons (0.15 m^3) of LN2 per day, so by connecting two Dewers together in series, one frame could run for over two days. However, liquid nitrogen supply problems from the manufacturer occurred during the test period resulting in several shut downs. Due to this, the lab's efficiency during the entire period dropped to approximately 60%. In comparison, NASA Langley's test frames ran close to 90% efficiency with the use of a 3,000 gallon (11 m^3) liquid nitrogen holding tank. In an effort to increase efficiency, CSULB installed a similar 3,000 gallon (11 m^3) holding tank and supply system. Since the installation of the tank, the laboratory is running at 95% efficiency.

Due to concerns with the frames going out of alignment over long periods of time, a data acquisition system capable of reading 12 strain gages and 4 thermocouples was installed in the laboratory. Twelve high temperature strain gages were adhesively bonded onto each panel. Six sets of back to back strain gages were installed in a cross configuration. Four thermocouples were also mounted in the corner regions of the panels that had in the past had a tendency to heat up at a slightly faster rate than the center of the panel. By installing these gages and thermocouples, the panel's mechanical and thermal strains could be monitored during the aging process prior to sacrificing each of the panels for residual strength testing.

The strain gauge results indicated no slippage or misalignment of the panels. Efforts were made to collect as much strain data as possible from the tests, however the long durability of the high temperature epoxy used to bond the strain gauges survived no more than 5,000 hours of testing during the second phase of testing. Despite the poor durability of the adhesive, the results from the strain gages and thermocouples did verify that the loads and temperatures seen in the panel's test section were indeed uniform.

Since the completion of the initial test program the lab has been accumulating over 7,000 hours per year per frame for seven frames since 1996. Keeping the laboratory running at 95% efficiency requires maintenance. The relays controlling the ovens require a large amount of servicing. Solenoid valves controlling the flow of liquid nitrogen had to be replaced on a regular basis. Mother boards to the Instron controllers also have a tendency to fail under these severe operating conditions. Having a supply of spares for each frame is crucial in operating this laboratory with a high degree of efficiency.

CONCLUSIONS

Boeing and California State University, Long Beach have recognized the need for additional dedicated facilities for long-term durability testing of candidate HSCT materials. CSULB has constructed a laboratory consisting of ten Instron load frames with eight thermal chambers that will be used to simulate the use environment of the HSCT. Testing will continue on the polyimide, but the cyanate ester has been withdrawn from the program due to its poor durability properties under these conditions. The polyimide has demonstrated little degradation in in-plane properties but out-of-plane properties have shown some early degradation, this may be due to some microcracking that is occurring in the material.

Maintenance issues associated with the laboratory must be addressed with a supply of spare parts. A staff must be available to monitor these tests continuously. Further checkouts of the material and equipment have been performed and verified that the materials are undergoing to loads generated by the equipment. Despite minor hang-ups in locating spares, the lab continues to run at 95% efficiency with a total of seven frames running in the lab generating 7,000 hours per frame per year.

REFERENCES

1. J.R. Kerr and J.F. Haskins, General Dynamics Corporation, Convair Division "Time-Temperature-Stress Capability of Composite Materials for Advanced Supersonic Technology Applications" , NASA Contractor Report 178272, Contract NAS1-12308